EQUILIBRIUM AND KINETIC STUDIES OF SYSTEMS OF HYDROGEN ISOTOPES, LITHIUM HYDRIDES, ALUMINUM, AND $LiAlO_2$

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ABSTRACT

Tritium might be bred by the ^{6}Li(n,α)T reaction in a solid lithium alloy or compound in the blanket of a controlled thermonuclear reactor to avoid problems associated with molten lithium or lithium compounds. Li-Al and LiAlO$_2$ systems containing hydrogen, deuterium, or tritium were studied 10-15 years ago at the Savannah River Laboratory. This paper describes measurements of (1) the distribution of tritium and helium throughout both α and β phases of irradiated Li-Al alloy, (2) the migration rate of tritium to the β phase during moderate heating, (3) equilibrium pressures as functions of temperature of H$_2$, D$_2$, or T$_2$ in contact with lithium hydrides + aluminum, Li-Al alloy, or irradiated Li-Al alloy, (4) the equilibrium constant for the reaction LiH + Al $\rightarrow$ LiAl + 1/2 H$_2$ as a function of temperature, and (5) extraction rates of tritium from irradiated LiAlO$_2$ targets at elevated temperatures.

INTRODUCTION

Tritium might be bred by the ^{6}Li(n,α)T reaction in a solid lithium alloy or compound in the blanket of a controlled thermonuclear reactor (CTR) to avoid problems associated with the use of molten lithium or a molten lithium compound. Lithium-aluminum and lithium aluminate systems containing hydrogen, deuterium, or tritium were studied 10 to 15 years ago at the Savannah River Laboratory (SRL).[*] Although these systems

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[*] Work done by L. H. Meyer, M. O. Fulda, D. W. Tharin, Jr., and A. A. Kishbaugh.

contained higher concentrations of hydrogen isotopes (1 to 80 cm^3 per cm^3 of alloy) than would be expected in a low-inventory blanket,[1] the data are applicable to CTR-oriented research.

Measurements at SRL have included:

1. Distribution of tritium and helium between the alpha and beta phases of irradiated Li-Al alloy.

2. Kinetics of tritium migration in the alloy.

3. Equilibrium conditions for the reaction between aluminum and LiH, LiD, or LiT.

4. Extraction rates of tritium from irradiated LiAlO$_2$.

## DISTRIBUTION OF TRITIUM AND HELIUM IN Li-Al ALLOY

In the 1.2, 3.2, and 7.4 wt % Li alloys of lithium and aluminum studied at SRL, an intermetallic compound, LiAl, is dispersed as a separate β phase in a dilute solid solution of Li in Al, the α phase. Figure 1 shows that at irradiation temperatures of ∿100°C, the solubility of Li in Al is low,[2,3,4] so that practically all of the Li is in the β phase. But because the recoil range of tritons (∿45 × 10^{-4} cm in the β phase, ∿30 × 10^{-4} cm in the α phase) is large compared to the size of the β-phase particles (radius ∿6.5 × 10^{-4} cm) and the distance between the particles (∿5 × 10^{-4} cm), the newly formed tritium is distributed throughout both α and β phases, depending in part on the relative volume of the β phase (24% for 3.5 wt % Li alloy, 47% for 7.4 wt % alloy). On the other hand, helium remains predominantly in the α-phase near β-phase particles, because the helium recoil range is about equal to the average distance between the particles.

Moderate heat treatment (150° to 375°C) promotes migration of both tritium and helium. Tritium is concentrated in the β phase, which acts as a sink for tritium because of the reaction

$$LiAl + T \rightarrow LiT + Al.$$
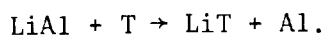
Helium tends to collect in gas pockets in the α phase, in close proximity to the β-phase particles where most of it was formed.

Distribution of tritium and helium, before and after heat treatment, was determined by sequential dissolution of small chips of alloy machined from an irradiated slug. About 15 to 20 mg were first exposed to methanol for ∿1 min, which dissolved much of the β phase:

$$2LiAl + 2CH_3OH \rightarrow 2LiOCH_3 + 2Al + H_2$$

$$LiT + CH_3OH \rightarrow LiOCH_3 + HT$$
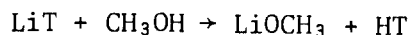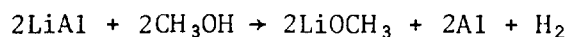
Both gas and liquid phases were analyzed for tritium, then the liquid was evaporated and the residue analyzed for lithium and aluminum. The un-dissolved residue from the methanol treatment (mostly the aluminum-rich α phase) was then totally dissolved in 6N HCl, and both gas and liquid phases were analyzed. Table 1 summarizes results of 13 separate tests.

The higher density of the aluminum-rich α phase, compared to the lithium-rich β phase, hinders diffusion of tritons. Therefore, as these dissolution tests show, the α phase of the 7.4 wt % Li alloy initially contains more tritium (∿81% of the total) than would be expected on the basis of its volume fraction in the alloy (53%). After heat treatment, however, most of the tritium is contained in the β phase.

These tests demonstrate that the helium content of the α phase, initially 70% to 90% of the total, is also diminished by heat treatment. The helium data are less reliable than the tritium data, because large corrections were required to allow for the solubility of helium in methanol and losses from gas pockets opened during machining and heat treatment.

KINETICS OF TRITIUM MIGRATION IN Li-Al ALLOY

The rate of migration of tritium to the β phase was measured by a second series of tests with 1/4" x 1" wafers, all cut from the same irradiated Li-Al alloy slug, then heat-treated before chips were removed for sequential dissolution and analysis. This technique reduces gas loss during heat treatment, so that the results summarized in Table 2 are probably more accurate than those for heat-treated chips in Table 1. Both sets of data are plotted in Figure 2.

The linearity of log % tritium in the α phase vs heat treatment time for 7.4 wt % Li wafers (Figure 2) indicates that the migration of tritium from the α to the β phase is a first-order reaction. Values of the rate constant k (sec^{-1}) as a function of temperature were computed from half-times read from these curves at 150°, 200°, and 300°C (Table 3). A plot of log k vs 1/T(°K) is approximately linear, yielding k = 2.35 × 10^{4} exp (-2.00 × 10^{4}/RT). The activation energy for the migration of tritium in the α phase is 20 ± 6 (std dev) kcal/mole.

Table 3. Measured Migration of Tritium from α
to β Phase in 7.4 Wt % Li Alloy

Temperature (°C)	Half-Time (sec)	Rate Constant k (sec^{-1})
150	1.26 × 10^{6}	5.50 × 10^{-7}
200	1.74 × 10^{4}	3.98 × 10^{-5}
300	2.00 × 10^{3}	3.47 × 10^{-4}

EQUILIBRIUM CONDITIONS FOR THE EXTRACTION OF HYDROGEN ISOTOPES

The reaction between LiT and aluminum,

$$LiT + Al \rightarrow LiAl + 1/2 T_2$$

is the mechanism for the release of tritium when irradiated Li-Al alloy is heated.* Figure 3 shows that around 500°C, the equilibrium pressure of tritium for this reaction is 300 to 600 times the pressure for the thermal decomposition of pure LiT.[8]

Tables 4 and 5 summarize SRL measurements of equilibrium pressures of hydrogen, deuterium, and tritium as a function of temperature; however, sufficient data to calculate equilibrium constants for the above reaction, as discussed below, were available for the 3.2 and 1.2 wt % alloys only (Table 5). Figure 4 is a schematic of the system used for these measurements.

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* Addition of lead has been found to reduce both H_2 and T_2 losses during extraction from LiH or LiT, apparently by combining with condensing lithium vapors at the cooler throat of the furnace, thereby preventing resorption of the hydrogen isotopes.[5] Lead flux reduces the amount of tritium retained in Li-Al alloy residues[6] and permits rapid gas evolution at ∿490°C.[7]

The alloys were maintained at a constant temperature; equilibrium was assumed to have been attained after the pressure remained constant for one hour. Equilibrium pressures were measured after both increasing and decreasing the temperature to the desired level; agreement was good except at lower temperatures where re-absorption was so slow that equilibration was impractical.

Equilibrium constants for the reaction with hydrogen were calculated as follows:

$$K = \frac{\alpha_{LiAl} \sqrt{\alpha_{H_2}}}{\alpha_{LiH} \alpha_{Al}}$$

where α_{H_2} = equilibrium pressure (mm)/760 mm atmos^{-1}, and where α_{LiH} = 1.00 was assumed because LiH exists as a separate phase in the binary Al-Li system.

Several assumptions are possible for the determination of α_{Al} and α_{LiAl}. First, α_{LiAl} might be taken as unity, because it exists as a separate β phase in the α-phase solid solution of Li and Al; and α_{Al} might be taken as $1 - \alpha_{Li}$, where α_{Li} is the mole fraction of the Li that is dissolved in the α phase at saturation, as computed from Figure 1.

But Figures 5 and 6 show that the equilibrium hydrogen pressure is a function of the hydrogen content of the Li-Al system (i.e., the "gas volume ratio"*). Therefore it would be better to assume that at high GVR's so much Li is converted to LiH that no LiAl exists as a separate β phase and even the α phase is not saturated with LiAl. Then the number of moles of LiAl in the α phase is not determined by saturation, but is the number of moles of Li originally present less the amount precipitated as LiH. At some intermediate GVR, all LiAl(β) has just been converted to LiH, and the α phase is just saturated with LiAl. Finally, for all lower GVR's, the α phase is saturated with LiAl, some LiAl is present in a separate β phase, and a small amount of LiH has been precipitated.

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* GVR = cm^3 of total gas/cm^3 of alloy.

However, this formulation does not fit the experimental data too well. First, the calculated equilibrium constants actually vary by nearly ±50%. Second, if the activity of LiAl remains constant as long as some LiAl is present as a separate phase (above the saturation curve, Figure 1), the hydrogen pressure must also remain constant: by the phase rule for 3 components (Li, Al, H) and 4 phases (LiAl + Al in α phase, LiAl in β phase, precipitated LiH, and H_2 gas), degrees of freedom = C - P + 2 = 3 - 4 + 2 = 1, which requires that the hydrogen pressure be invariant at any given temperature. This invariance is not observed even at the lowest GVR values (Figures 5,6).

Since greater and greater pressures are required to force additional increments of hydrogen into the sample, regardless of whether or not a separate LiAl phase is present, it appears that LiAl *behaves* as if it were always dissolved in the α phase (then C - P + 2 = 3 - 3 + 2 = 2 degrees of freedom, temperature *and* pressure variable). This could occur if the activity of the LiAl decreases with increasing formation of LiH. Table 5 shows equilibrium constants calculated according to this last formulation. For example, in 1 cm^3 of 3.2 wt % Li alloy (density = 2.46 g/cm^3) at 375°C,

$$n_{Al}^o = n_{Al(\alpha)} + n_{LiAl(\alpha)} = \text{moles of free Al + Al in LiAl, in } \alpha \text{ phase}$$

$$= \frac{(2.46 \text{ g alloy})(0.968 \text{ g Al/g alloy})}{26.98 \text{ g/mole Al}} = 0.0883$$

$$n_{Li}^o = \text{total number of moles of Li}$$

$$= \frac{(2.46 \text{ g alloy})(0.032 \text{ g Li/g alloy})}{6.94 \text{ g/mole Li}} = 0.0113$$

$$n_{LiAl(\alpha)} = n_{Li}^o - n_{LiH}$$

$$= n_{Li}^o - \frac{GVR}{11200 \text{ cm}^3/\text{g atom hydrogen}}$$

$$\alpha_{LiAl} = \frac{n_{LiAl(\alpha)}}{n_{LiAl(\alpha)} + n_{Al(\alpha)}} = \frac{n_{LiAl(\alpha)}}{n^\circ_{Al}}$$

$$= \frac{n^\circ_{Li} - (GVR/11200)}{n^\circ_{Al}} = \frac{0.0113 - (GVR/11200)}{0.0883}$$

$$\alpha_{Al} = 1 - \alpha_{LiAl}$$

Then since $\alpha_{LiH} = 1.00$ and $\alpha_{H_2} = \sqrt{P_{H_2}}$, atmos,

$$K = \frac{\alpha_{LiAl}}{\alpha_{Al}} \sqrt{P_{H_2}} = \frac{127 - GVR}{862 + GVR} \sqrt{P_{H_2}} \text{ for 3.2 wt \% Li alloy.}$$

Similarly, for 1.2 wt % Li alloy, density = 2.58 g/cm^3,

$$n^\circ_{Al} = 0.0945$$

$$n^\circ_{Li} = 0.00446$$

$$\alpha_{LiAl} = \frac{n^\circ_{Li} - (GVR/11200)}{n^\circ_{Al}} = \frac{0.00446 - (GVR/11200)}{0.00945}$$

$$\alpha_{Al} = 1 - \alpha_{LiAl}$$

$$K = \frac{\alpha_{LiAl}}{\alpha_{Al}} \sqrt{P_{H_2}} = \frac{50.0 - GVR}{1008 + GVR} \sqrt{P_{H_2}} \text{ for 1.2 wt \% Li alloy.}$$

It may seem inconsistent to assume that the LiH phase is completely separate from the Li + LiAl matrix, whereas the LiAl phase is considered to be dissolved. But LiH is chemically much more like a salt than a metal and would not be expected to dissolve in aluminum. Furthermore, equilibrium constants computed on this basis (Table 5) are now constant within about ±7%.

A plot of log K vs 1/T, Figure 7, yields the entropy and heat of reaction:

LiH + Al → LiAl + 1/2H$_2$ ΔH = 14.9 ±0.4 (std dev) kcal/mole

ΔS = 14.5 ±0.6 (std dev) cal/mole deg
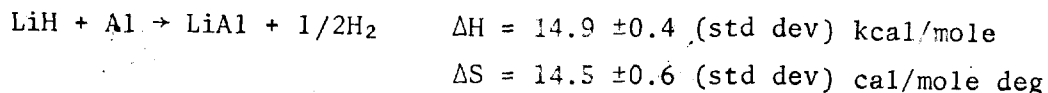
The heat of formation of LiAl calculated from this value and from

the heat of formation of LiH[10] is -6.8 kcal/mole, in comparison with a reported value of -13.0 ±4.0 kcal/mole.[11]

Data were insufficient for similar calculations of equilibrium constants for deuterium and tritium.

EXTRACTION OF TRITIUM FROM IRRADIATED LITHIUM ALUMINATE

In 1966, SRL developed a process for the extraction of tritium from irradiated $LiAlO_2$ targets.[12] The New Production Reactor at Hanford required tritium-producing targets that would not melt even if the zirconium-clad targets were heated to ∿1100°C. Lithium aluminate was chosen as the most suitable target material (m.p. = 1900°C) after tests of its irradiation stability. It had been proposed to extract tritium from declad $LiAlO_2$ targets by dissolution in molten borax at 850°C. But experiments at SRL showed that tritium could be extracted by simply heating the targets at 850°C in a vacuum for 11 hours, leaving <0.1% of the tritium in the residue; borax flux was not only unnecessary, but increased tritium losses to 1-2%.

Typical gas evolution rates are shown in Figures 8 and 9. Table 6 shows how tritium losses can be reduced by prolonged heating and/or higher temperatures.

In these tests, hydrogen isotopes were separated from other extraction gases by Hopcalite* (catalytic oxidizer) and zeolite (water absorber) beds in series, followed by a uranium bed decomposer (Figure 10). After loading the furnace and pumping the system down to <50 microns, the furnace was heated and evolved gases were passed through the Hopcalite bed at 500°C to oxidize hydrogen isotopes. Water vapors were sorbed on the zeolite bed at 50°C while remaining gases (mainly CO, CO_2, and He) passed through and were discharged. After completion of extraction, the zeolite was heated to 500°C and desorbed water vapors were passed through the uranium decomposer bed at 550°C to regenerate the gaseous hydrogen isotopes.

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* Tradename of Mine Safety Appliances Co.; 80:20 mixture of MnO_2, CuO_2.

Additional experiments showed that the presence of CO and CO_2 equivalent to 10-30 volume % of the tritium did not degrade the performance of the uranium decomposer. No CH_4 was formed, which would have decreased the permeability of the palladium diffuser used in subsequent operations to separate hydrogen isotopes from ^{3}He and ^{4}He; the hydrogen permeation rate at 450°C did not change during exposure of the palladium diffuser to a mixture of H_2, CO, and CO_2 for >1700 hr. A Hopcalite-zeolite system to separate CO and CO_2 from hydrogen isotopes appears to be unnecessary for a full-scale extraction process.

Table 1. Distribution of Tritium and Helium in Irradiated Li-Al Alloy

Wt % Li in Alloy	Chips from Slug No.	Heat Treatment Time (hr)	Heat Treatment Temp (°C)	Fraction of β-Phase Dissolved by CH_3OH (%)	Fraction of Total Tritium in α Phase (%)[a]	Fraction of Total Helium in α Phase (%)
7.4	1		None	79.6	71.9	83
	1		None	78.2	84.4	89
	2		None	70.4	85.5	72
	2		None	76.2	83.4	76
	2	1	200[b]	77.3	0.0	37
	2	1	300[b]	79.2	0.0	44
	1	4	375[c]	74.5	0.0	74
	1	4	375[c]	70.6	3.5	-
	1	4	375[c]	73.4	3.8	59
3.5	3		None	16.0	25	81
	4		None	15.5	73	93
	4		None	16.7	84	90
	4	1	300[b]	28.7	40	66

[a]Corrected for incomplete dissolution of β phase by CH_3OH.

[b]Chips heat-treated after machining.

[c]Slug heat-treated before machining.

Table 5. Equilibrium Constants for the Reaction LiH + Al → LiAl + 1/2H₂

Wt % Li in Alloy	Temp (°C)	$\frac{1000}{T(°K)}$	GVR (cm³ H₂/cm³ alloy)	P_{H_2}[a] (mm)	$\sqrt{P_{H_2}}$ (atm)$^{\frac{1}{2}}$	α_{LiAl}	$\alpha_{Al} = 1 - \alpha_{LiAl}$	K
3.2	375	1.543	2.5	10	0.1147	0.1260	0.8740	0.0165
			5	11	0.1203	0.1234	0.8766	0.0169
			10	12	0.1257	0.1184	0.8816	0.0169
			20	15	0.1405	0.1082	0.8918	0.0171
			30	18	0.1539	0.0981	0.9019	0.0167
			40	22	0.1701	0.0880	0.9120	0.0164
			50	27	0.1885	0.0779	0.9221	0.0159
			60	32	0.2052	0.0678	0.9322	0.0149
			70	49	0.2539	0.0577	0.9423	0.0155
			80	89	0.3422	0.0476	0.9524	0.0171
	475	1.337	2	143	0.4338	0.1265	0.8735	0.0628
			5	160	0.4588	0.1234	0.8766	0.0646
			10	175	0.4799	0.1184	0.8816	0.0644
			20	230	0.5501	0.1082	0.8918	0.0668
			30	295	0.6230	0.0981	0.9019	0.0678
			40	380	0.7071	0.0880	0.9120	0.0682
			50	500	0.8111	0.0779	0.9221	0.0685
			60	640	0.9177	0.0678	0.9322	0.0667
1.2	325	1.672	5	6	0.0889	0.0425	0.9575	0.0039
			10	9	0.1088	0.0378	0.9622	0.0043
			20	23	0.1740	0.0283	0.9717	0.0051
			30	67	0.2969	0.0189	0.9811	0.0057
			40	240	0.5620	0.0094	0.9906	0.0053
	375	1.543	5	52	0.2616	0.0425	0.9575	0.0016
			10	80	0.3244	0.0378	0.9622	0.0127
			20	180	0.4867	0.0283	0.9717	0.0142
			25	270	0.5960	0.0236	0.9764	0.0144
	395	1.497	5	140	0.4292	0.0425	0.9575	0.0191
			10	200	0.5130	0.0378	0.9622	0.0201
	460	1.364	1	530	0.8351	0.0463	0.9537	0.0405
			3	710	0.9665	0.0444	0.9556	0.9449

[a]Hydrogen pressures corresponding to specified GVR's were read from Figures 5 and 6.

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FIGURE LIST

Fig. 1. Solubility of Lithium in Aluminum (Solid Phase).

Fig. 2. Time Dependence of α Phase Tritium in Heat-Treated 7.4 wt % Li Alloy.

Fig. 3. Equilibrium Pressures of Hydrogen Isotopes for the Decomposition of Lithium Hydrides With and Without Aluminum.

Fig. 4. Schematic Diagram of Apparatus for Measuring Pressures of Hydrogen Isotopes in Equilibrium with Reaction Mixtures.

Fig. 5. Hydrogen Pressure-Concentration Isotherms for 3.2 Wt % Li Alloy.

Fig. 6. Hydrogen Pressure-Concentration Isotherms for 1.2 Wt % Li Alloy.

Fig. 7. Temperature Dependence of the Equilibrium Constant for the Reaction $LiH + Al \rightarrow LiAl + 1/2H_2$.

Fig. 8. Effect of Time and Temperature on Extraction of Tritium from Irradiated $LiAlO_2$.

Fig. 9. Gas Evolution from Irradiated $LiAlO_2$ at 900°C Without Borax Flux.
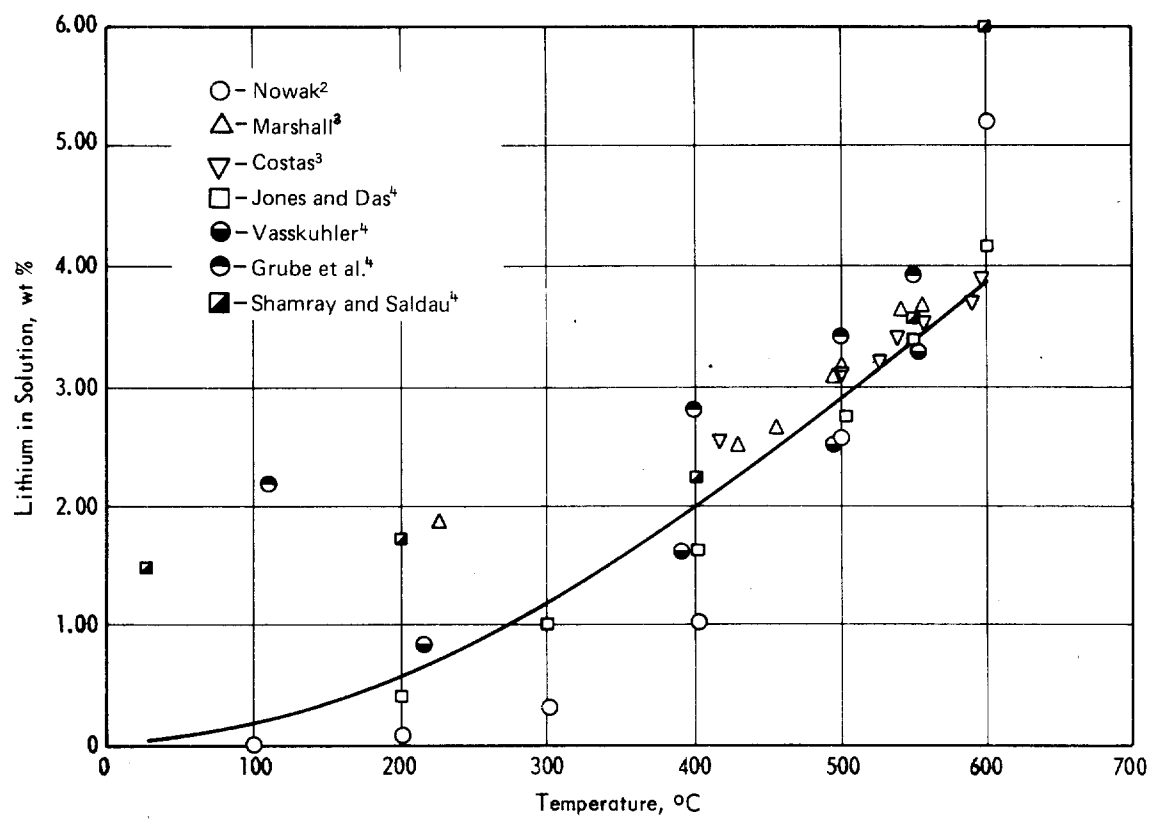
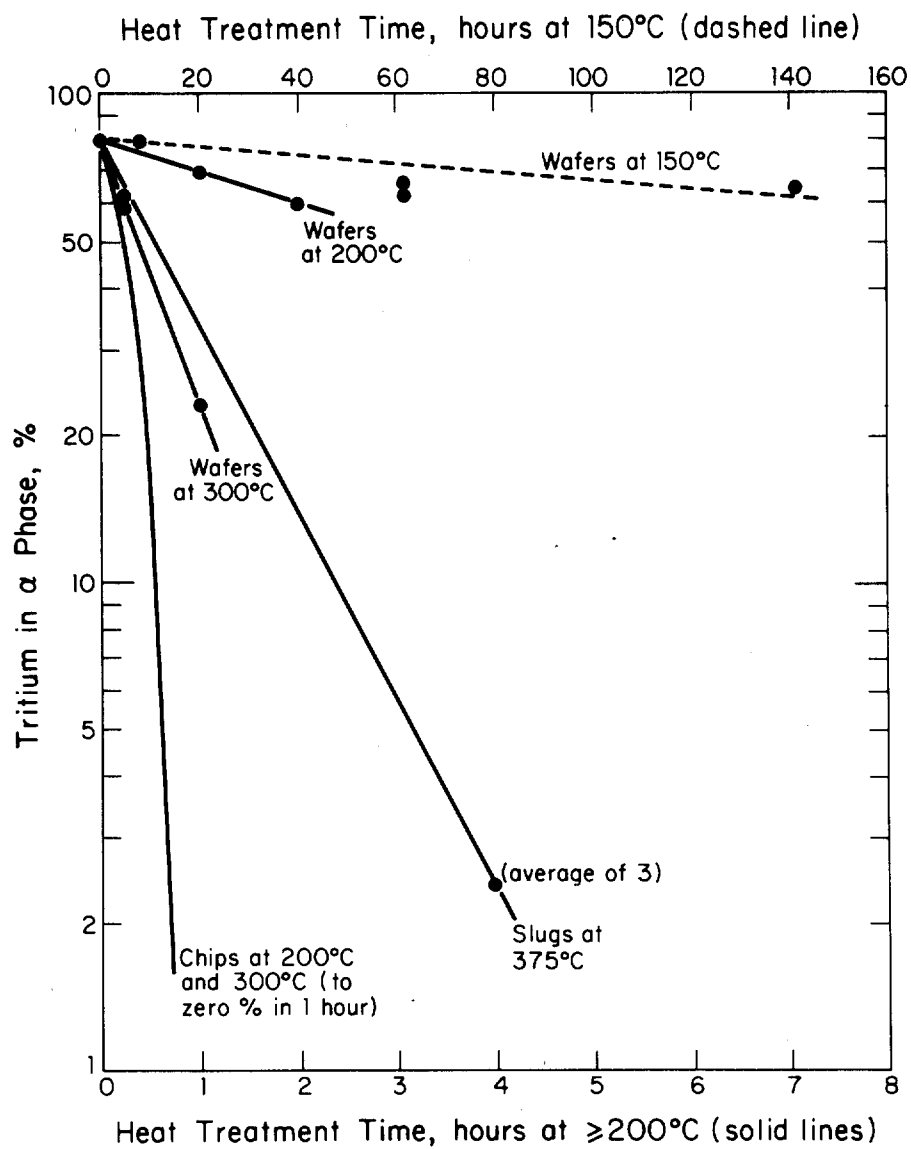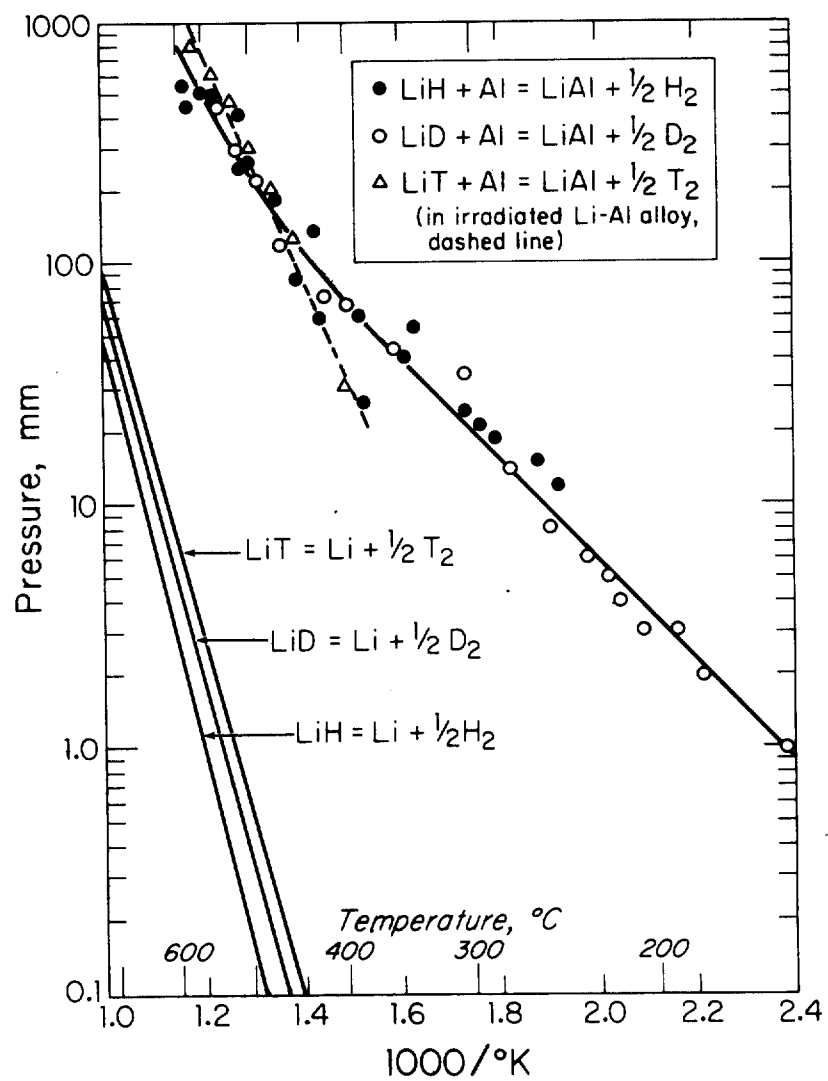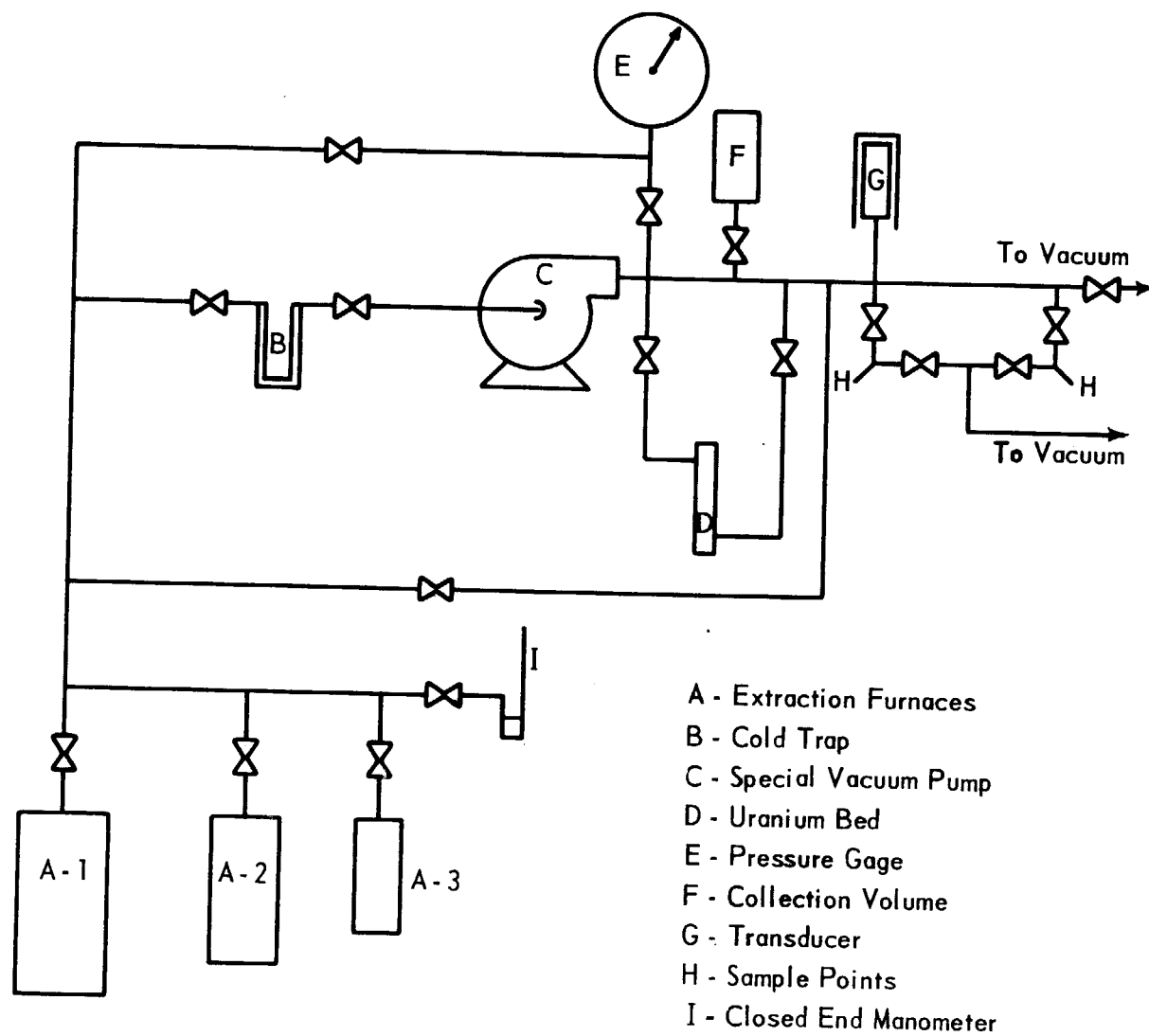Fig. 10. Experimental Extraction Facility.

FIG. 1

Heat Treatment Time, hours at 150°C (dashed line)

Tritium in α Phase, %

Wafers at 150°C

Wafers at 200°C

Wafers at 300°C

Chips at 200°C and 300°C (to zero % in 1 hour)

(average of 3)

Slugs at 375°C

Heat Treatment Time, hours at ≥200°C (solid lines)

FIG. 2

FIG. 3

To Vacuum

To Vacuum

A - Extraction Furnaces
B - Cold Trap
C - Special Vacuum Pump
D - Uranium Bed
E - Pressure Gage
F - Collection Volume
G - Transducer
H - Sample Points
I - Closed End Manometer

FIG. 4

FIG. 5

FIG. 6

$$Ln\ K = -\frac{\Delta H}{RT} + \frac{\Delta S}{R}$$
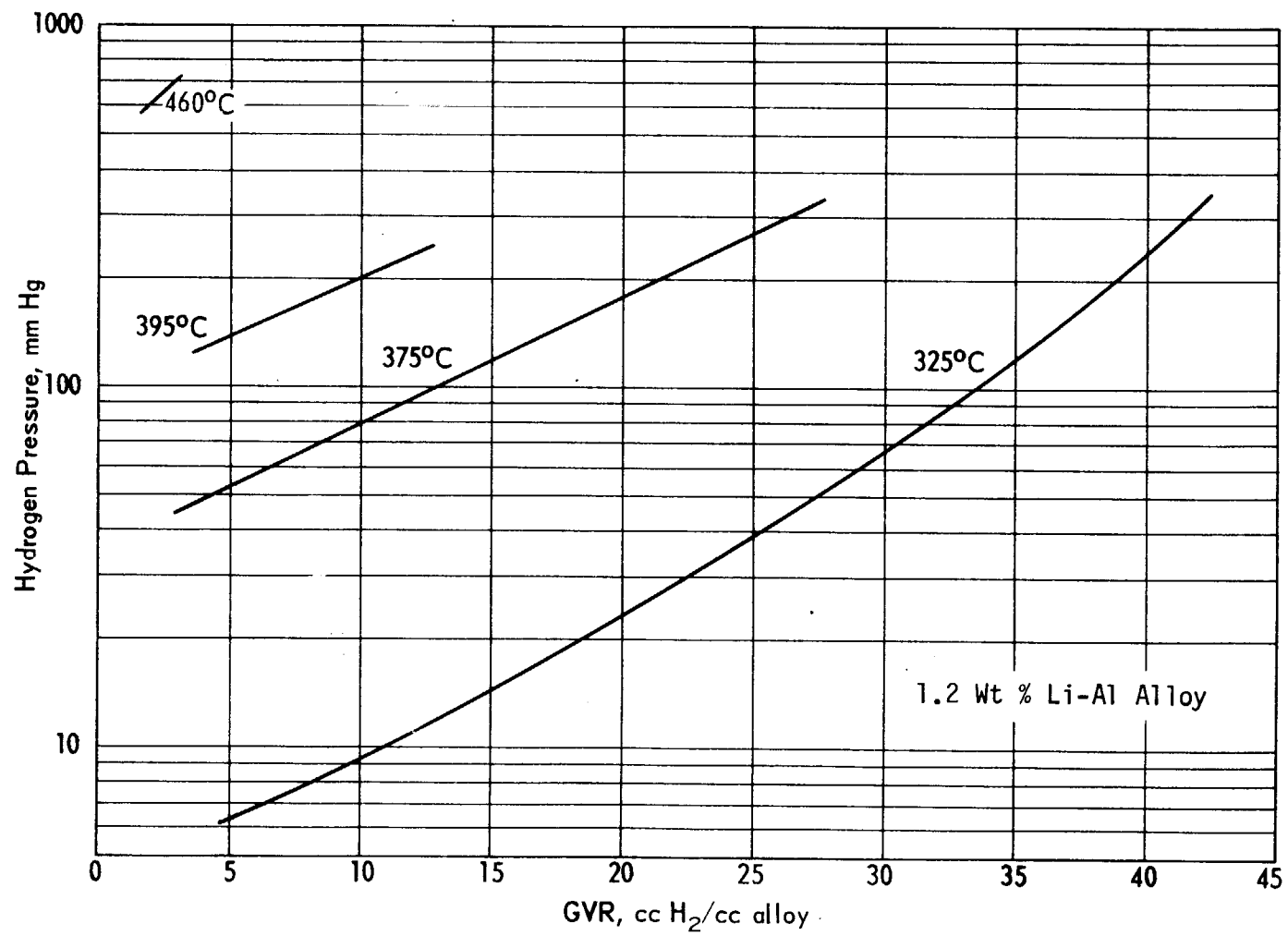
$\Delta H = 14.9 \pm 0.4$ kcal mole^{-1}

$\Delta S = 14.5 \pm 0.6$ cal mole^{-1} deg^{-1}

Temperature, °C

FIG. 7

FIG. 8

FIG.4

A - Extraction Furnace
B - "Hopcalite" Bed
C - Cooling Water Jacket
D - Zeolite Bed
E - Cold Trap

F - Uranium Bed Decomposer
G - Special Vacuum Pump
H - 0-150 cm Hg Ashcroft Gage
I - Closed End Manometer
J - 8339-cc Collection Vessel

K - 33.2-cc Collection Vessel
L - 77.6-cc Collection Vessel
M - Modified Welch "Duo-Seal"
     Vacuum Pump
N - Sample Point

To
Stack
Or
Uranium
Storage
Bed

FIG. 10